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Wear Properties of Carbon Nanotubes Filled Epoxy Polymers and Woven Glass Fiber Reinforced Polymer Composites

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ABSTRACT

This research investigated the wear properties of Carbon Nanotube (CNT) filled epoxy polymer and fiber reinforced composites. The CNT/epoxy composites with 0.5 wt% and 1.0 wt% CNT contents were mixed at 50°C for 1 hour at a speed of 400 rpm using mechanical mixer, while woven glass fiber reinforced polymer (GFRP) nanocomposites were fabricated using vacuum bagging technique. The effect of CNT on wear properties was evaluated using dry sliding abrasion wear test that used vitrified bonded silicon carbide as abrasive wheels. The mass loss and specific wear rate curves show that wear properties of epoxy polymer and GFRP composite systems were enhanced when CNT was added. Epoxy polymer and GFRP nanocomposites showed the highest wear resistance when CNT content was 1.0 wt% and 0.5 wt% respectively. The CNT-filled composite showed improvement till up to 78.9 % from its pure system. This suggested that the load transferability between CNT and epoxy was more effective in nanomodified systems than in its pure systems. Therefore, adding CNT improves the wear properties of epoxy polymer and woven GFRP composite.

Keywords: CNT, dry abrasion, epoxy, glass fiber, specific wear rate

INTRODUCTION

In recent years, the demand for advanced composite materials has increased due to their availability and their utility in many applications. Wear performance of a material is based on the material properties in terms of its wear rate and friction coefficient. It can be defined as the loss of material when subjected to relative motion, and it further includes any form of surface damage caused by rubbing processes. Thus, composite materials embed

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reinforcement into the matrix to improve the properties of the material. Epoxy resin matrix, a thermoset type polymer, has been used extensively in engineering applications, due to its flexible processing characteristics, good affinity to heterogeneous materials, considerable creep and solvent resistance as well as its high operating temperatures (Guo et al., 2010). However, there are still limitations to using epoxy matrix, such as brittleness, rigidity, and poor resistance to crack propagation (Mirmohseni & Zavareh, 2010). Therefore, fillers and fibers have been reinforced to formulate high mechanical and wear resistant composite materials. The fillers have been developed from micro-size to nano-size today to give a better impact on the matrix. Nano-size fillers are added for better stiffness, strength, toughness, dimensional stability, and thermal properties (Jumahat et al., 2013). Fillers such as carbon nanotubes (CNTs) have high aspect ratio (Meng et al., 2009) and outstanding mechanical, electrical, thermal, and other characteristics, making them a multi-functional reinforcement material for polymer matrices (Sapiai et al., 2015; Kim et al., 2012).

Fiber reinforced polymer composites hold extra credit because of their lightweight, excellent specific stiffness and strength, and liberty in design due to tailored anisotropy behaviour (Chen et al., 2014). Glass fibers on the other hand, is a remarkable material due to their high strength to weight ratio and high corrosion and chemical resistance (Kanny & Mohan, 2014), besides their wide availability as well as being inexpensive (Zhao et al., 2013). However, the properties of FRP composite depend on several factors such as fiber orientation, fiber content, fiber length, fiber/matrix compatibility and its interface strength (Guignier et al., 2015; Sapiai et al., 2015).

Many studies have been conducted in the past decade on CNT (nanofillers) reinforced polymer composites and glass fiber reinforced polymer focusing on their mechanical and tribological properties. While for advanced type composite structure where fiber serves as primary reinforcement and nanofiller as secondary reinforcement, studies on tribological properties have are new (the past 5 to7 years). A study by Suresha et al. (2010) looked at carbon and glass fabric reinforced vinyl ester composites, where a test was conducted using block on ring dry wear test to find out wear behaviour of the composites. The result showed that carbon fiber showed better wear resistance than glass fiber, due to the abrasiveness of the latter that ruptured transfer film and adhesive bonds at the interface of the composite.

Larsen et al. (2008) examined glass and carbon/aramid weave epoxy composite using pin on disc to determine wear performance at 9 different p-v conditions. It was found that glass/epoxy is beneficial if fibers in the weave are oriented parallel and anti-parallel with respect to sliding direction instead of normal and parallel. The findings are also affected by fiber properties, as reported by (Suresha et al., 2010). Dong et al. (2005) studied the tribological properties of nanocomposites where 0-4.0 wt% MWCNT content was incorporated into epoxy matrix and tested under dry sliding contact condition. It was found that MWCNTs/EP composites have higher wear resistance and also smaller friction coefficient compared with pure epoxy system. Nanocomposites with 1.5wt% MWCTNs content showed smallest wear rate and friction coefficient. Yan et al. (2013) on the other hand had studied the performance of aligned CNT (ACNTs) epoxy composite when undergoing water-lubricated sliding contact.

They concluded that first, ACNTs improved wear resistance of ACNTs/EP composites by 219 times than pure epoxy, and second, water lubricant improved wear mechanism from fatigue wear to slightly abrasive wear.

Although enhanced mechanical properties of CNT filled polymer composite have been extensively investigated, the wear properties of advanced nano-filled fiber reinforced composite have not yet been fully studied. In the present work, wear behaviour (mass loss and specific wear rate) of CNT-filled epoxy and CNT-filled Glass Fiber Reinforced Polymer (GFRP) composites under abrasive condition was studied. Sample characterisations such as TEM, density, heat distribution and optical micrograph were conducted.

METHOD

In this work, the epoxy resin (Miracast 1517 A) and hardener (Miracast 1517 B) used in this research were supplied by Miracon (M) Sdn. Bhd; the mixing ratio was 100:30. A FloTube 9000 Series Multi-wall Carbon Nanotube (CNT) was used as nanofiller, supplied by CNano Technology (Beijing) Ltd, China. The CNT was produced by a catalytic vapour deposition process with average diameter and length of 11 nm and 10 μm respectively. The glass fiber used was CWR200 plain weave C-glass fiber supplied by Vistec Technology Service (M) Sdn. Bhd.

The desired amount of CNT (0.5 wt%, and 1.0 wt%) was measured and mixed in epoxy resin using mechanical stirrer at 50°C temperature and speed of 400 rpm for 1 hour. The mixture was then degassed under high vacuum oven for 1 hour to remove oxygen and air bubbles. Finally, the mixture was blended with hardener for 15 minutes before used.

For CNT-filled epoxy system, the samples were prepared using silicon mould. The silicon mould was prepared following dimension shown in Figure 1. The surface of silicon mould was coated with release agent to ease sample removal once it cures. Curing process was done at room temperature for 24 hours. For CNT-filled GFRP composite system, vacuum bagging method was used to laminate glass fiber and CNT-filled epoxy resin. The CNT-filled epoxy resin of 0.5 wt% and 1.0 wt% was prepared by following the exact procedure mentioned above. 24 sheets of glass fiber was layered with mixture of CNT-filled epoxy resin to ensure the 6mm sample thickness, as required by machine specification. Vacuum process was done for 1 hour to remove trapped air. Finally, the laminates were cured at room temperature for 24 hours. The cured laminates were cut based on the dimension shown in Figure 1, in accordance with machine specification.

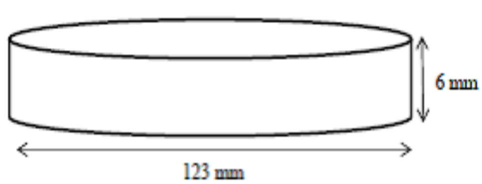


Figure 1. Dimensions for dry sliding abrasion test

The degree of CNT dispersion in epoxy matrix was observed using Transmission Electron Microscopy (TEM). The sample was cut into a thickness of 85nm using Leica UC2 Ultra-microtome machine at room temperature. The TEM machine used was FEI Tecnai TEM at accelerating voltage of 80 kV. Gatan MS6000CW high-resolution digital camera was used to capture the image, while Gatan digital micrograph software was used to collect the image with magnifications of 43000x and 300000x.

The density of sample was determined by Archimedes' principle using density balance. The size of the composite sample was 15 mm x 15 mm x 3.0 mm and calculated in accordance to ASTM D792. The density was obtained following Eq. [1]. Measurement was repeated 5 times for each sample system.

$$\rho_{sample} = \frac{A}{A-B} \times \left[\left(\rho_{water} - d \right) + d \right] \qquad [1]$$

Where ρ_{sample} is density of sample, ρ_{water} is density of water at room temperature (20°C) = 0.9982 g/cm^3, A is mass of dry sample and B is mass of sample in water.

Abrasion Resistance Tester (TR-600) was used to conduct dry sliding abrasion wear test. The schematic diagram of the tester is shown in Figure 2. The test was conducted based on ASTM D3389 standard. The disc of 123 mm diameter and 6 mm thickness was the sample, located in contact with two vitrified bonded silicon carbide as the abrasive wheels. In each test run, the wheels were cleaned using metal brush to eliminate any dust, particle and contaminant. The weight of sample at initial stage and after each run was taken using high precision balance. The sample was set at 267 rpm speed and 20 N load for 10,000 m distance travel with interval of 2000 m. A total of five readings of mass was taken for 10,000 m distance travel. Based on the mass loss recorded, the specific wear rate was calculated using Archard's wear model (Archard, 1953), stated in Eq. [2]. The summary of operational conditions is given in Table 1.

$$Ws = \frac{\Delta m}{L \times \rho \times F} \qquad [2]$$

Where W_s in (mm^3/Nm), Δm is mass loss (g), L is sliding distance (m), ρ is density (g/mm^3) and F is applied load (N).

The thermal distribution at the contact surface was captured using Infrared camera (PTI 160) to obtain the amount of heat generated during testing. To further investigate the worn surface, optical microscopy was done using Stereo-zoom Microscope. This was facilitated using IMAP software, where the software is able to enlarge the image of sample in order to investigate the scratch and wear mechanisms on the surface.

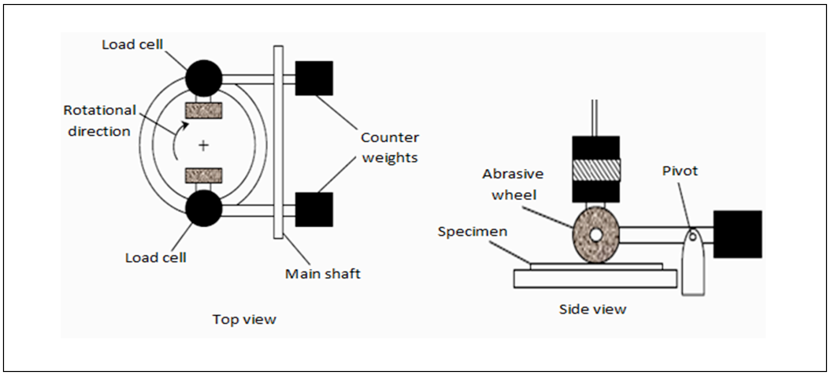


Figure 2. Schematic diagrams of Abrasion Resistance Tester (TR-600) (Jumahat et al., 2015)

Table 1
Operating parameters of Dry Sliding Abrasion Test

Parameters	Experimental Conditions
Contact geometry	Cylinder on flat
Type of motion	Unidirectional sliding
Applied load	20 N
Sliding speed	267 rpm
Sliding distance	10,000 m at interval 2000 m

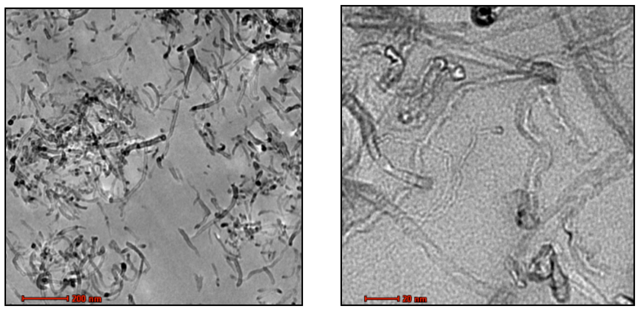
RESULTS AND DISCUSSION

Based on Eq. (1), the density of CNT-filled epoxy and CNT-filled GFRP composite was obtained, shown in Table 2. The density of epoxy filled with CNT was increased as CNT content increased while CNT-filled GFRP composite showed different trend as CNT content increased. Although fiber reinforcement did increase the density of composite, the latter decreased as CNT content increased. This may be due to the presence of voids in the composite and uneven dispersion of CNT inside the matrix. The density further affects the result of specific wear rate of samples, as shown in Eq. (2).

Table 2
Density of sample

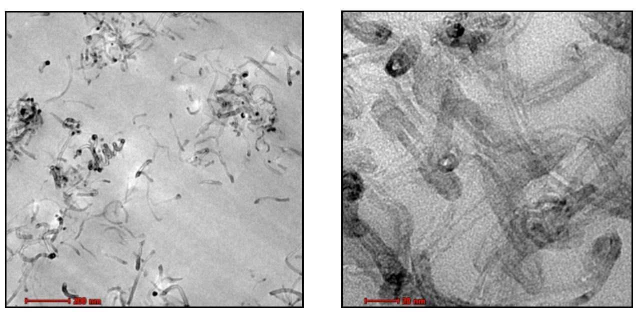
Epoxy content (wt%)	CNT content (wt%)	Glass Fiber content (vol%)	Density of composite (g/cm^3)
100.0	-	-	1.14850
99.5	0.5	-	1.14981
99.0	1.0	-	1.14998
100.0	-	10.0	1.65552
99.5	0.5	10.0	1.48892
99.0	1.0	10.0	1.41586

The dispersion of 0.5 wt% and 1.0 wt% CNT in epoxy resin is shown in Figure 3 and Figure 4 respectively. At high magnification, TEM images showed the presence of clusters of entangled CNT in epoxy. The CNT has an average diameter of 10 nm as shown in high magnification TEM images.



(a) (b)

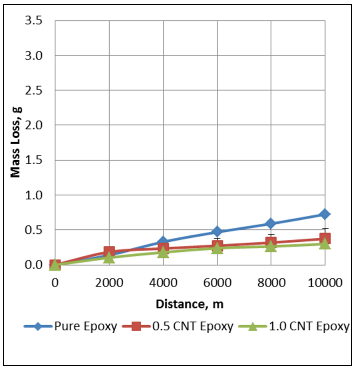
Figure 3. TEM images for dispersion of 0.5 wt% CNT-filled epoxy at magnification of: (a) 43000x; and (b) 300000x

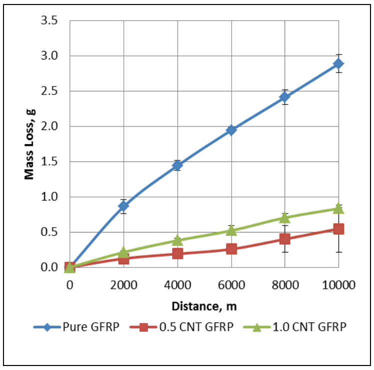


(a) (b)

Figure 4. TEM images for dispersion of 1.0 wt% CNT-filled epoxy at magnification of: (a) 43000x; and (b) 300000x

The results for accumulated mass loss of CNT-filled epoxy and CNT-filled GFRP composite are shown in Figure 5(a) and Figure 5(b) respectively. The results were compared with pure epoxy and pure GFRP composite system. The mass loss over 10,000 m distance follows a similar increasing trend for all samples of pure epoxy, pure GFRP composite, CNT-filled epoxy and CNT-filled GFRP composite. In Figure 5(a), the accumulated mass loss for pure epoxy, 0.5 wt% CNT and 1.0 wt% CNT-filled epoxy composites are 0.725 g, 0.325 g and 0.300 g respectively. The 1.0 wt% CNT-filled epoxy showed lowest mass loss, improving by 58.2 % from epoxy without nanofillers. It can be observed that the CNT incorporation in epoxy has reduced the amount of accumulated mass loss of the samples, although the improvement was not that significant.



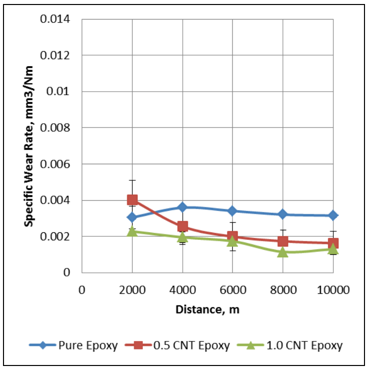


(a) (b)

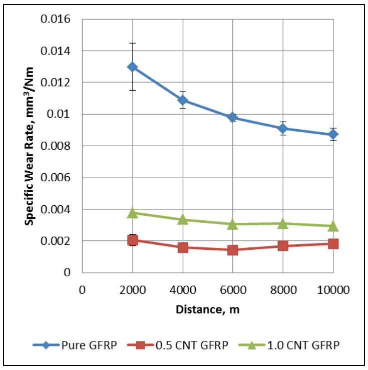
Figure 5. Curves of accumulated mass loss versus distance for: (a) CNT-filled epoxy; and (b) CNT-filled GFRP composite when compared to pure system using dry sliding abrasive test

In Figure 5(b), the profile trend for 0.5 wt% and 1.0 wt% CNT-filled GFRP composite have shown great improvement compared with that of pure GFRP composite. The lowest mass loss is 0.547 g from sample with 0.5 wt% CNT content. Both samples of 0.5 wt% and 1.0 wt% CNT-filled GFRP composite have shown wear improvement up to 81.1% and 71.1% respectively. However, pure GFRP composite has highest accumulative mass loss up to 2.887 g, very high compared with pure epoxy in Figure 5(a). This is due to the presence of glass fiber in the composite. Glass fibers are abrasive and brittle in nature (Suresha et al., 2010). Its debris may form third-body abrasive wear, leading to higher mass loss (Larsen et al., 2008). Moreover, 1.0 wt% CNT-filled GFRP composite performance was not at par as 1.0 wt% CNT-filled epoxy performance, might be due to agglomeration of CNT in the matrix. However, by observing both figures, the results actually proved that resins incorporated with nanofillers do improve its wear resistant compared with their pure system.

From accumulated mass loss values, corresponding specific wear rate profiles after 10,000 m distance travel were plotted in Figure 6 using Eq. (2) and the density results obtained before. In Figure 6(a), the specific wear rate after 10000 m is highest for pure epoxy compared with 0.5 wt% and 1.0 wt% CNT-filled epoxy composites with a value of 0.003156 mm^3/Nm. The lowest specific wear rate is using 1.0 wt% CNT-filled epoxy composite with a value of 0.0013044 mm^3/Nm, indicating its high wear resistance. The percentage improvement was about 56.67 % from pure epoxy. Furthermore, by observing the three profiles, the presence of CNT showed improvement on the composite, although the improvement only occurred after 4000 m distance travel. This is due to the run-in wear stage or initial wear stage that begins from 0 m to 4000 m distance, and above 4000 m, the composite has achieved its steady-state stage (Lee et al., 2014).



(a)



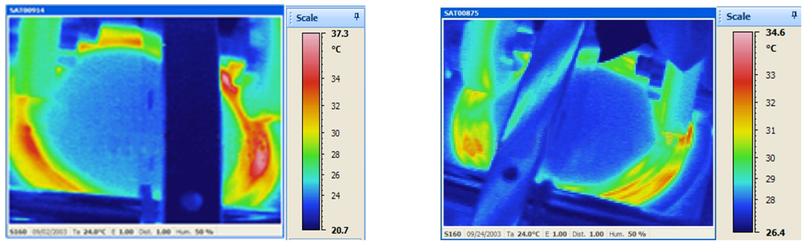
(b)

Figure 6. Curves of specific wear rate versus distance for: (a) CNT-filled epoxy; and (b) CNT-filled GFRP composite compared with pure system using dry sliding abrasive test

For the case of CNT-filled GFRP composite, shown in Figure 6(b), the specific wear rate profiles for pure, 0.5 wt% and 1.0 wt% CNT content showed a more consistent improvement. As described in Figure 5(b), 0.5 wt% CNT-filled GFRP composite has the highest wear performance, thus the lowest specific wear rate with a value of 0.0018358 mm^3/Nm, improving by 78.9% compared with pure GFRP composite. The sample with 1.0 wt% CNT content has shown less wear improvement that may be due to nanofiller agglomeration (Dong et al., 2005).

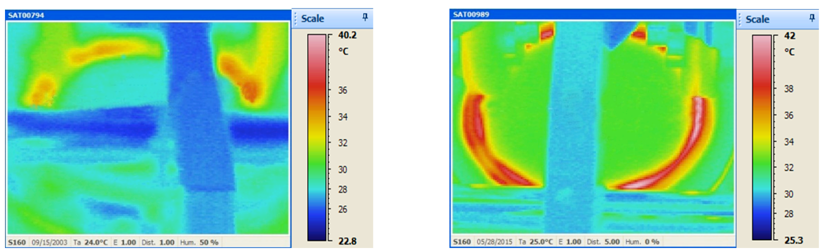
Figure 7 and Figure 8 show thermal distribution of the contact at the wheel for both systems after 6000 m distance travel, for CNT-filled epoxy system and CNT-filled GFRP composite system respectively. The maximum temperature recorded for CNT-filled epoxy system was 37.3°C and 34.6°C for 0.5 wt% and 1.0 wt% CNT content respectively. The thermal distribution showed that the frictional heat for 1.0 wt% CNT-filled epoxy is lower, which resulted in lower wear rate. On the other hand, higher maximum temperature was recorded for CNT-filled GFRP composite systems. 0.5 wt% CNT content recorded temperature of 40.2°C while 1.0 wt% CNT content recorded even higher temperature up to 42°C. The frictional heat created on the surface of the latter sample was very high, resulting in a higher mass loss, complying with the low specific wear rate discussed before. Besides that, the presence of glass fiber contributed to the frictional heat due to the abrasive nature of glass fiber (Suresha et al., 2010).

The worn surface of pure GFRP composite and CNT-filled GFRP composite for dry sliding abrasion test after 10,000 m distance travel was selected to evaluate the effect of CNT content on the worn surface, as shown in Figure 9.



(a) (b)

Figure 7. Thermal distribution of: (a) 0.5 wt%; and (b) 1.0 wt% CNT-filled epoxy composite

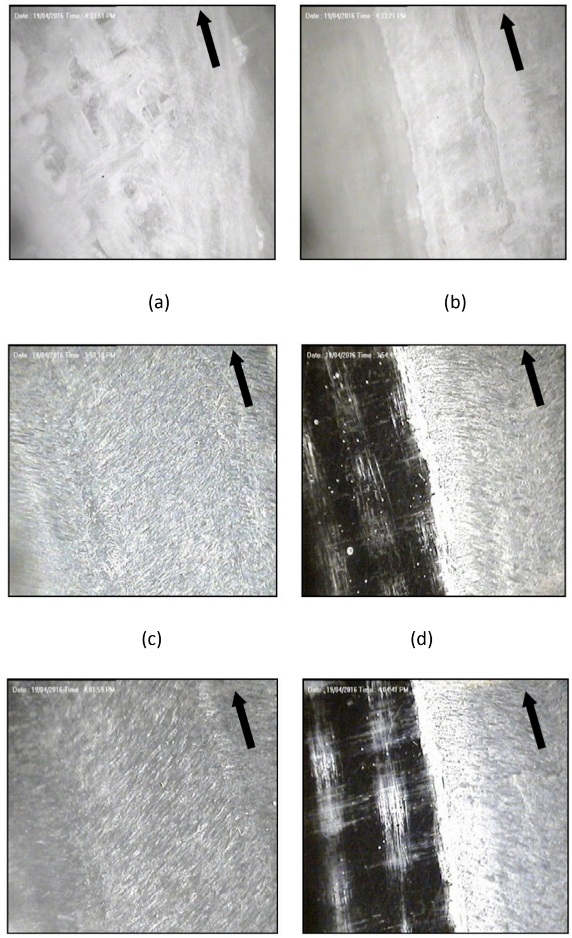


(a) (b)

Figure 8. Thermal distribution of: (a) 0.5 wt%; and (b) 1.0 wt% CNT-filled GRFP composite

The worn surface of samples exhibited different morphologies due to different interfacial strengths. The worn surface of pure GFRP composite shows signs of adhesion and abrasive wear (Figure 9(a)). The surface displayed plucked and ploughing marks indicating adhesive wear and ploughing. The damage starts with microcracks leading to resin removal. Once the fiber is not protected by resin, they become exposed to fracture due to their brittle and fragile behaviour (Larsen et al., 2008). The boundary of wear track and unworn surface (Figure 9(b)) was not easily distinguished due to the high resin removal. The matrix/fiber interface strength was not strong, corresponding with the result obtained in Figure 5(b).

Incorporating CNT in GFRP composite showed improvement in worn surfaces as displayed in Figure 9(c and d) and Figure 9(e and f). No fiber pull out or ploughing was found in either samples. The surface was also smoother, uniform and compact, without any microcracks of resin. The adhesive wear was clearly reduced, as proven by results in Figure 5(b). The boundaries can also be distinguished easily due to the strong interface strength between fiber and matrix. Therefore, incorporating CNT into epoxy matrix reduces wear of GFRP composites.



(a) (b) (c) (d) (e) (f)

Figure 9. Worn surface and track boundary of dry sliding abrasion test for: (a and b) Pure; (c and d) 0.5 wt% CNT-filled; and (e and f) 1.0 wt% CNT-filled GFRP composite

CONCLUSION

This study had examined the wear properties (mass loss and specific wear rate) for CNT-filled epoxy and CNT-filled GFRP composite under dry sliding abrasive test. It was found that the wear properties of pure epoxy and pure glass fiber reinforced composite were improved when CNT is used as a filler. For nano-filled epoxy, CNT content of 1.0 wt% have the lowest specific wear rate with 56.67% improvement from pure epoxy system, while for nano-filled glass fiber reinforced composite, composite with 0.5 wt% of CNT have the lowest specific wear rate, with improvement of 78.9% from pure glass fiber reinforced composite. The presence of CNT improved the interface strength between fiber and matrix for fiber reinforced composite system. In order to further investigate the wear resistance of these systems, it is recommended future studies focus on the effect of operating parameter during testing.

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